U.S. Department of Commerce
C. William Verity
Secretary

National Bureau of Standards
Ernest Ambler, Director

National Bureau of Standards

Certificate of Analysis

STANDARD REFERENCE MATERIAL 482

Gold-Copper Wires for Microprobe Analysis

These standard reference materials are designed for use in quantitative elemental microprobe analysis. Although the selection of this particular system was circumscribed by the requirements of standard reference materials for electron probe microanalysis, the materials will be equally useful for other micro techniques. Accurate chemical characterization and the achievement of homogeneity on a microscopic scale were given special emphasis.

SRM 482 wire	Color code	Nominal composition	Cominco American[a]	U.S. Bureau of the Mint[b]		NBS[c]		Average value[d]	
			Au	Au	Cu	Au	Cu	Au	Cu
			Percent by weight						
A	Gold	Au100	–	–	–	–	–	100.0_0	–
B	Gray	Au80 – Cu20	80.10	80.13	19.81	80.21	19.85	80.1_5	19.8_3
C	Yellow	Au60 – Cu40	60.30	60.37	39.66	60.41	39.62	60.3_6	39.6_4
D	Blue	Au40 – Cu60	40.12	40.06	59.88	40.11	59.97	40.1_0	59.9_2
E	Red	Au20 – Cu80	20.04	20.12	79.84	20.21	79.86	20.1_2	79.8_5
F	Copper	Cu100	–	–	–	–	–	–	100.0_0

[a] The fire assay method was employed for the determination of Au by Cominco American.

[b] At the U. S. Bureau of the Mint, Au was determined by fire assay and Cu was determined by electrodeposition.

[c] At NBS, Au was determined by precipitation from solution; Cu was determined by electrodeposition.

[d] The results of individual laboratories agree within a range of ± 0.1% absolute from the average values. The agreement between results by the different methods and analysts, and the summation of results close to 100% for each binary alloy, indicate that the averages are free from significant bias.

The set of standard reference materials, SRM 482, consists of six wires each having a diameter of approximately 0.5 mm and a length of approximately 5 cm. For identification, the four alloy wires were covered with an easily removable colored coating.

The overall direction and coordination of technical measurements leading to certification were performed under the chairmanship of B. F. Scribner.

The technical and support aspects involved in the preparation, certification, and issuance of these standards were coordinated through the Office of Standard Reference Materials by R. E. Michaelis.

Gaithersburg, MD 20899
August 25, 1988
(Revision of certificate dated 6-6-69)

(over)

Stanley D. Rasberry, Chief
Office of Standard Reference Materials

PREPARATION AND PURITY: The standards were prepared by Cominco American, Inc. in the form of wires approximately 150 meters long. The end members of the series, as well as the starting materials for the alloys, were of the highest purity grade and precautions were taken to minimize contamination. Two of the alloy standards were heat-treated at NBS to improve microhomogeneity. The pure metal standards were examined by the residual resistivity ratio technique and the total of electrically active impurities in each was estimated to be about 0.001%. The gold-copper wires were examined spectrographically for metallic impurities; no significant impurities were found at detection limits ranging from 0.0001 to 0.010%.

LONGITUDINAL HOMOGENEITY: Variation in composition along the full length of each alloy wire was investigated by electron probe microanalysis for areas 25 μm diameter on cross sections at three positions along the wire including the two ends. The observed differences in composition for the positions, expressed as the range between the highest and lowest values for each alloy, were as follows:

Nominal Composition	Au80	Au60	Au40	Au20
Observed range*	0.3%	0.7%	0.9%	0.9%

Homogeneity along the wires was also tested by measurement of the residual resistivity ratio. These measurements indicated that the variation (macroscopic) of composition along all standard wires was less than 0.1% absolute. Further information on longitudinal homogeneity of the wires was obtained by determinations of Au at the extreme ends of the alloy wires by the Bureau of the Mint; the data also indicate that the extreme variation along the wires is less than 0.1% absolute.

TRANSVERSE AND MICRO HOMOGENEITY: Variation in composition within the above mentioned cross sections of the wires was investigated by electron probe microanalysis. For each cross section, measurements were made along two diagonals at right angles. On each diagonal, determinations were made at 25 points, 1 μm or less in diameter, starting and ending at approximately 25 μm from the edge. For each alloy, the element which could be determined with the better precision was used in the evaluation. The variation was calculated in terms of the standard deviation for an individual determination for each traverse. In the table below, the variation is presented as the range between the lowest and highest observed standard deviations for the six traverses performed on each alloy.

Nominal Composition	Element Determined	Range of Standard Deviations for Traverses*
Au80	Cu	0.09 – 0.24%
Au60	Cu	.16 – .27
Au40	Au	.13 – .23
Au20	Au	.13 – .20

The homogeneity on a microscopic scale was further investigated by performing quantitative measurements in two arrays of 10 × 10 points (1 μm diameter) on each of the cross sections. The distance between adjacent points was 3.5 μm. This was repeated on several cross sections so that 6 arrays were obtained on each alloy. For the element which could be measured with better precision, the range is given between the lowest and highest observed standard deviation for an individual determination for the 6 arrays for each alloy.

Nominal Composition	Element Determined	Range of Standard Deviations for Arrays*
Au80	Cu	0.19 – 0.28%
Au60	Cu	.28 – .37
Au40	Au	.25 – .31
Au20	Au	.12 – .20

*The ranges indicated are close to the precision of the method and should be considered upper limits of estimates of inhomogeneity.

Extensive homogeneity studies were performed with the electron probe microanalyzer at NBS by M. A. Giles, D L. Vieth, R. L. Myklebust, C. E. Fiori, and K. F. J. Heinrich. Measurements of residual resistivity ratio were made at NBS, Boulder, Colorado, by R. L. Rutter and R. L. Powell. Heat treatment of the alloys at NBS was performed by G. E. Hicho and M. R. Meyerson. Spectrographic survey analyses were made at NBS by V. C. Stewart. Determinations of composition were made at Cominco American, Inc., Spokane, Washington, by T. A. Rice; at the U. S. Bureau of the Mint, Washington, D. C., by H. G. Hanson, Jr.; and at NBS by J. R. Baldwin and R. A. Durst.